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(71) Applicant *(for all designated States except US)*: ECO CORPORATION [CA/CA]; 6725 Airport Road, Suite 502, Mississauga, Ontario L4V 1V2 (CA).

(72) Inventors; and
(75) Inventors/Applicants *(for US only)* : WARRINGTON, John, E. [CA/CA]; 36 Alder Road, Toronto, Ontario M4B 2Y4 (CA). FORSBERG, Erik, B. [SE/CA]; 518 Gordon Baker Road, Willowdale, Ontario M2H 3B4 (CA). AUKSI, Hillar [CA/CA]; 22 Harmon Avenue, Aurora, Ontario L4G 4G6 (CA).

(74) Agents: NASSIF, Omar, A. et al.; McCarthy Tetrault, P.O. Box 48, Toronto Dominion Bank Tower, Toronto, Ontario M5K 1E6 (CA).

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(54) Title: OIL SPILL CLEAN-UP MATERIAL

(57) Abstract

An oil spill clean-up material capable of selective sorption of oil from water, especially crude oil from seawater, is comprised of a chopped, compacted foam polyethylene, shredded into a web-like, mesh condition, and exhibiting a highly porous, microcavernous network structure. It can be prepared from scrap polyethylene foam or from polyethylene foam prepared from either virgin or recycled polyethylene, by chopping, compacting to destroy the foam structure, and then shredding the compacted foam pieces.

* See back of page

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OIL SPILL CLEAN-UP MATERIAL

TECHNICAL FIELD

This invention relates to an oil sorbing material, and processes for its manufacture and use. More particularly, it relates to a polyethylene based oil sorbing material particularly adapted for sorbing crude oil spilled on seawater.

BACKGROUND ART

Whilst the need for emergency measures and materials to cope promptly and efficiently with oil spills on land and water has been apparent for some time, and many procedures and materials have been proposed and employed for this purpose in the past, the recent disaster of a massive oil spill off the coast of Alaska has highlighted the fact that workable solutions to these problems still do not exist. The problem becomes particularly acute when the spill occurs in a remote area and under inhospitable weather conditions.

An important ingredient in any oil spill cleanup operation is cost. Cleanup materials will not likely be prepared ahead of time and kept in convenient storage locations close to disaster-prone areas if they are too expensive. Speed of reaction to an oil spill disaster is normally very important to any reasonably successful containment and cleanup thereof. Accordingly, any sorbent material which is to be used should be inexpensive to produce, and easy and inexpensive to transport.

Polyethylene has long been recognized as an oil sorbent material which is relatively cheap and easy to transport. It has been proposed for use in oil spill

cleanup operations in a variety of different forms. For example, Canadian patent 1,091,217 (Bucheck et al) describes fibrillated films of polyolefin as a fluffy, bulky product to sorb large volumes of high viscosity oil. More specifically, it describes an oil sorbing mat having sections of fibrillated film which has been fibrillated, i.e. provided with a myriad of small parallel cuts, and expanded, i.e. stretched, to at least four times its original width transverse to the direction of the cuts, to form a lightweight net-like structure. Several of these sections are superposed over one another and attached together. Whilst several different polymers are mentioned as suitable, only polypropylene is specifically exemplified. Canadian Patent 1,010,016 (DeYoung) describes sorption material in cellular form and impregnated with a hydrophobic and oleophilic sealant, to reduce the water absorbance. The material may be crushed and shredded, but retains its cellular nature. Whilst polyurethane foam is proposed as the best material, polyethylene is also mentioned as a possible foam.

A particular difficulty with oil spill cleanups from marine areas is the requirement to sorb the heavy, highly viscous components of the crude oil. Few materials have adequate sorption capacity for heavy viscous oils. Moreover, if an oil spill is not treated quickly, the lighter oil fractions tend to disperse and evaporate, so that the resultant residue is more concentrated in these heavy viscous fractions.

DISCLOSURE OF THE INVENTION

It is an object of the present invention to

provide a novel, economic oil sorption material and a process for its preparation.

Accordingly, the present invention provides polyethylene in web-like, mesh form, prepared by compacting foamed polyethylene pieces, substantially to destroy the typically closed cell foam structure, and then shredding it. It exhibits a microcavernous network structure. The material need contain no sorption additives, binders, sealants or the like so that it is extremely inexpensive. Moreover, it can be prepared from very inexpensive starting materials, including scrap foam polyethylene, which is made in enormous quantities for use in packaging. Huge quantities of off-cuts of these materials are currently being discarded and used as landfill, creating serious disposal problems. The process for preparation of the materials according to the invention is simple and economical to conduct, and can be run continuously and at high speed.

The polyethylene products of this invention show an outstanding ability to sorb oil from oil-water mixtures, both fresh water and salt water. They are effective not only in sorbing light, relatively low and medium viscosity hydrocarbon oils, but also high viscosity heavy oils such as bunker C oil and number 5 fuel oil. They retain reasonable strength upon sorption so that they can be withdrawn along with the sorbed oil after use. Additionally, it is believed that the polyethylene products of this invention are able to sorb light hydrocarbons such as gasoline from hydrocarbon-water mixtures.

Thus, according to one aspect of the present invention, there is provided a process for preparing polyethylene sorbent material useful for sorbing oil spilt on water, which comprises:

compacting small foam polyethylene pieces so as largely to destroy any closed cell foam structure thereof and to open substantially all of the foam cells thereof to interior liquid access;

and shredding the compacted foam pieces to a web-like, microcavernous network condition, effectively completing the destruction of the closed cell structure.

According to another aspect of the present invention, there is provided a process for selectively sorbing liquid hydrocarbons from water, which comprises applying to the hydrocarbon/water mixture a web-like polyethylene material having a microcavernous network structure prepared from foamed polyethylene pieces by a process of compacting the pieces largely to destroy the cellular structure thereof, then shredding the compacted pieces to a network form.

From a third aspect of the present invention, there is provided a sorbent material for sorbing liquid hydrocarbons, and comprising polyethylene pieces having a web-like, microcavernous network structure.

Whilst it is not intended that the invention be limited to or bound by any particular theory or mode of action, it is believed that the microcavernous structure of the crushed, shredded foam material of the present invention is likely to be largely responsible for the oil sorptive properties thereof. In general, the materials of the invention are made up of thin section polyethylene pieces, or flakes, of a thickness

consistent with those normally encountered in cell wall thicknesses of closed cell polyethylene, exhibiting microcaverns of a diameter and depth no bigger than the cell sizes commonly encountered in closed cell polyethylene foams. Typically the openings of the microcaverns are 0.01-2.5 mm in size, and the depths of them are essentially similar. The materials of the present invention are not, however, limited to any particular size range in respect of their microstructure. The microstructure of the materials is in sharp contrast to that exhibited by competitive polyolefin sorbents currently on the market, such as that marketed by 3M as 3M-T240, which has a fibre-like microstructure.

BRIEF DESCRIPTION OF THE DRAWINGS

Embodiments of the invention will be described with reference to the accompanying drawings, in which:

FIGURE 1 is a diagrammatic process flow sheet illustrating a preferred embodiment of a process according to the invention;

FIGURE 2A is a microphotograph of a section through a piece of closed cell foamed polyethylene, at a magnification of 20 times;

FIGURE 2C is a microphotograph of a section through a piece of shredded, crushed, foamed polyethylene according to the invention, at a magnification of 20 times;

FIGURE 3 is a graphical presentation of the results of Example 2 given below; and

FIGURE 4 is a graphical presentation of the results of Example 3 given below.

BEST MODE FOR CARRYING OUT THE INVENTION

With reference to FIGURE 1, irregularly shaped lumps 10 of polyethylene closed cell foam, after separation of other scrap residues therefrom, are fed to a chopper 12 in which they encounter rotary cutting elements 14 adjusted to cut and chop the lumps 10 into convenient small-sized pieces 16, suitably of size about 0.5 inches (12.7 mm). The pieces 16 are then fed by conveyor 18 into a compactor 20 in which the pieces 16 are subjected to a relatively severe compaction process by passage through the nip between press rollers 22. The separation between the rollers 22, and the load which can be applied to them, is adjustable. The pieces 16 are compacted to such an extent that substantially all the cells therein are broken open and the cellular foam structure is effectively totally destroyed. All surfaces of the material, including those formerly comprising inner cell surfaces, are then available for liquid contact.

Next, the material, in compacted piece form, is fed by conveyor 24 into a shredder 26 where it encounters rotary blades 28 which shred the compacted pieces into a web-like material 30. The material 30 is then packaged using conventional means, ready for shipping to the oil spill location.

The material 30 has a generally fluffy, flaky appearance, similar to that of light, fluffy snow. On closer examination under magnification, each piece is seen to be microcavernous network but in the form of an interconnected fibrous web-like irregular lace, rather than individual fibres. It is soft and somewhat resilient to the touch. The remnants of cells can be observed, but as microcaverns in the polyethylene pieces. No intact closed cells are observable even under magnification.

With reference to FIGURE 2A of the accompanying drawings, this shows the microstructure of conventional closed cell foamed polyethylene, constituting the starting material for the preferred product of the present invention. This microphotograph is taken on freshly sectioned material. It shows irregularly shaped cells with essentially continuous walls indicating its closed cell nature. Typically cell diameters are 0.5-1.5 mm.

Almost all foamed polyethylene currently manufactured has a closed cell structure. It may have a density of from 0.5-10 lbs./ft.^{3} (8.01-160.2 kg/m^3), preferably 1-4 lbs./ft.^{3} (16.02-64.08 kg/m^3). The source of foam polyethylene is not particularly restricted. For example, scrap or virgin foam polyethylene may be used. Alternatively, scrap polyethylene may be converted into a foam which is of relatively low quality for most applications but is completely suitable for use to prepare the polyethylene products of the invention.

FIGURE 2B of the accompanying drawings similarly shows the microstructure of the foamed

polyethylene material after the crushing operation, effected by compactor 20. This microphotograph is taken on freshly sectioned material, to show the cross-section in the direction of crushing. The deformation of the cells, as compared with those shown in FIGURE 2A, indicates that they do not relax completely after crushing. The numerous holes and tears in the cell walls show that the closed cell structure has been largely destroyed.

FIGURES 2C and 2D of the accompanying drawings show similarly the microstructure of the preferred product according to the invention, i.e. after shredding. The destruction of the closed cell nature of the material has been completed by the shredding process, to yield a "snowflake-like" material with a microcavernous, irregular-edged structure.

Throughout the process of manufacture, the material is maintained in a dry, free-flowing form so that it is easily and economically handled, throughout the chopping, transporting, compacting and shredding steps. The process as shown can be run continuously and at high speeds, and the starting material is a very inexpensive, often scrap, commodity, so that the end product can be made very inexpensively indeed. It is safe to handle, non-irritant, and dense enough to be handled easily by gravity feed, in open apparatus.

The material produced according to the invention shows an outstanding affinity for oil when the oil is mixed with water. When added to a vessel containing an oil and water mixture, the material soaks up the oil in a matter of less than 2-3 minutes, and remains strong enough to be removed from contact with

the water whilst retaining most of the oil it has absorbed. Most of the oil can be pressed and squeezed out of the material, to render the material suitable for re-use, should this be desired. Alternatively, the material containing oil can be safely incinerated. The water can be fresh water of salt water. The material exhibits very strong preference to absorb the oil from the water.

Moreover, the material absorbs both light oil and heavy oils. These heavy oils include crude unrefined oils of the type spilled as a result of oil tanker accidents at sea, bunker C oil, diesel oils, motor lubricating oils and even contaminated sump oils, of the type which might be discharged from a marine engine after use. The materials will absorb many times their own weight of any of these hydrocarbons, i.e. up to 25 times their own weight, in a matter of minutes.

In practice, the materials may be enclosed in water- and oil-resistant pervious containers such as nylon mesh bags, for ease of containment and withdrawal of the material after use. They may be used in conjunction with containment booms and other apparatus applied to marine oil spill cleanup. Further, the may be used in cleaning up oil spills on land or in machine shops and the like. They may also be used to assist in the cleanup of living objects, animals, birds and sea creatures which have become coated or otherwise contaminated with the oil. For this purpose, it is preferred to allow the living object to exercise in an enclosure containing the clean-up material in a loose fluff/flake form. The material itself is harmless on contact with living tissue, fur, feathers, shells, and the like.

The invention is further described, for illustrative purposes, in the following specific examples which should not be construed as limiting the scope of the invention.

EXAMPLE 1 -- DETERMINATION OF THE EFFICIENCY OF THE ABSORBENTS IN PICKING UP MOTOR OIL

A sorbent material was prepared as described above using the process outlined in FIGURE 1, and using as starting material a 2 lb./ft.^{3} (32.04 kg/m^3) closed cell polyethylene foam. Samples of the material so produced, and a 3M-T240 absorbent, were placed in vinyl screen bags which served to contain the absorbents, while allowing oil and water to pass through freely. The ability of the two materials to pick up and retain motor oil (10-W-30) was measured.

Two experiments were carried out. In the first, 0.90 g samples of the absorbents were added to a water bath containing 40 g of motor oil. The absorbents were allowed to become saturated with oil (about 10 minutes). The samples were then removed and allowed to drain until there was one oil drop per 15 second interval. The time for the oil drip rate to drop to 1 drop/15 seconds and weight of oil retained at that point was recorded. The results are shown in Table 1. It was found that the two absorbents had similar capacities, while the 3M-T240 reached the 1 drop/15 seconds slightly faster. Duplicate runs were made. The values given in Table 1 represent the average of two runs.

In the second experiment, the samples were treated the same way except that the samples were allowed to drain for 30 seconds and then weighed. In this case, the sorbent according to the invention showed a significantly higher holding capacity than 3M-T240.

Table 1

Absorbent Material	EXPERIMENT 1 Weight of Oil (grams) after draining until 1 drop/15 sec.	Drain time (min.) until 1 drop/15 sec.	EXPERIMENT 2 Weight of Oil (grams) abs. after 30 sec. of draining
Sorbent of the invention	11.4 ± 0.8	15.5 ± 0.5	20.0 ± 0.4
3M T240	11.7 ± 0.2	10.5 ± 0.5	14.2 ± 0.7

EXAMPLE 2 -- DETERMINATION OF THE EFFICIENCY OF THE ABSORBENTS FOR PICKING UP HEAVY CRUDE OIL

A sorbent material was prepared as in Example 1 but using a 3 lb./ft.^{3} (48.06 kg/m^3) closed cell polyethylene foam as starting material. It was evaluated as a sorbent for heavy crude oil, namely Wolf-Lake Crude Oil 22° API, in comparison with the fibrous 3M-T240 absorbent.

1 g of each of the absorbents was accurately weighed and stuffed in to a square screen pillow (6.5 cm x 6.5 cm approximate size) and heat sealed. The pillow was dipped into the heavy crude oil for the given time period. The oil soaked pillow was then removed from the

crude oil using tweezers and allowed to drain in a glass funnel for 10 minutes. The weight of the drained pillow was measured. The pillow was dipped into heavy crude oil for another period of time, drained for 10 minutes and the pillow weight measured. This process was repeated until 90 minutes of absorption time was completed. From the data, the amounts of oil absorbed (in g) per g of the absorbent for various absorption periods could be obtained, as shown in Table 2. The samples were allowed to drain for 10 minutes prior to weighing. The graph of heavy oil absorption (Figure 3) indicated that both the absorbents reached maximum absorption in 30 minutes. However, it is obvious that the absorbent of the invention can pick up more heavy crude oil than the 3M absorbent.

Table 2

Efficiency of Absorbents in Picking up Heavy Crude Oil (Wolf Lake Crude)

Absorbents	Absorption Time	Weight of Heavy Crude Oil Absorbed
Absorbent of the invention	5 min.	15.62 g
	10 min.	18.71 g
	30 min.	23.34 g
	90 min.	25.55 g
3M-T240	5 min.	13.09 g
	10 min.	15.96 g
	30 min.	18.32 g
	70 min.	18.05 g

EXAMPLE 3 -- DETERMINATION OF THE EFFICIENCY OF ABSORBENTS IN PICKING UP LIGHT CRUDE OIL (MSW)

Samples prepared as described in Example 2, from the same starting materials, were tested as previously described except that instead of heavy crude oil a light crude oil (MSW) was used. The weights of the absorbed oil at various absorption times are given in Table 3.

The corresponding graph (FIGURE 4) showed that for this batch of absorbent the 3M absorbent is more efficient than the absorbent according to the invention in picking up light crude oil, but both are efficient. Both the absorbents reached maximum absorption of light crude oil in 30 minutes.

Table 3

Efficiency of Absorbents in Picking Up Light Crude Oil (MSW)*

Absorbents	Absorption Time	Weight of Light Crude Oil Absorbed
Absorbent of the invention	5 min.	5.95 g
	10 min.	8.42 g
	30 min.	8.70 g
	90 min.	8.35 g
3M-T240	5 min.	10.53 g
	10 min.	11.39 g
	30 min.	11.93 g
	90 min.	12.06 g

*The samples were allowed to drain for 10 minutes prior to weighing.

EXAMPLE 4 -- CLEANUP OF MICE SOAKED WITH LIGHT CRUDE OIL

Three plastic tanks were filled with the same amount of water and light crude oil (MSW). Three mice of the same weight were used in this test.

One of the mice was put into the first tank. After swimming for three minutes, the oil-soaked mouse was taken out and was killed in an ether-filled jar. Then it was extracted with hexane, after the evaporation of which the amount of oil absorbed by the mouse was estimated. 7.8 g of crude oil was absorbed on the mouse.

In the second tank, the second mouse was allowed to swim for three minutes. Then excess absorbents according to the invention were added to cover the oil and then the mouse was allowed to swim 5 more minutes. It was taken out and the amount of oil left absorbed on the mouse was estimated as above. 2.1 g of crude oil was left absorbed.

In the third tank, the last mouse was allowed to swim for three minutes. Then the oil-soaked mouse was allowed to move around in a container half filled with absorbent according to the invention. After about 5 minutes, the mouse looked quite clean and was removed to estimate the residual oil content left absorbed on the mouse. 0.9 g of crude oil was left absorbed.

EXAMPLE 5 -- CLEAN UP OF MICE EXPOSED TO A SIMULATED OIL SPILL

Twelve mice (six male and six female) having a body weight in the range 20-30 g were divided into two

groups of six mice each and allowed a seven day acclimatization period. One group was assigned as a Test (cleaned up) Group while the other was assigned as a Control (noncleaned) Group. Plastic containers (26 cm x 20 cm x 15 cm) with a surface area of 520 cm^2 were filled with water at 20-22°C. 104 g (0.2 g/cm^2 surface) of crude oil was spilled over the surface of water in the containers.

Each of the six mice in the Test Group were placed into a separate container with the simulated oil spill. The mice were allowed to swim in the oil spill for two minutes. Thereafter, the mice were lifted from the container and each mouse was shaken gently to allow loose droplets of water/oil to drip off. The animals were then placed into a large glass container with a perforated lid and 40 g of absorbent according to the invention. The jar was slowly rotated for 10 minutes to allow the mice full exposure to the absorbent. Thereafter, the mice were placed in their respective cages for further observation.

The mice in the Control Group underwent exactly the same procedure as the mice from the Test Group with the exception that the mice in the Control Group were not exposed to the absorbent of the invention. Thus, after exposure to the simulated oil spill, the mice in the Control Group were placed directly into their respective cages for further observation.

Food and water were supplied to both Groups of mice as they were observed daily for fourteen days.

None of the mice in the Test Group displayed any sign of toxic effect after exposure to the simulated oil spill, with the exception of a very slight weight loss for the first three days after exposure. Essentially, all mice in the Test Group appeared clinically normal by the second day of observation after exposure to the simulated oil spill.

By contrast, the mice in the Control Group exhibited initially slight body weight loss, and some animals display toxic symptoms such as apathy, petosis, hunched back and cachexia. By the sixth day after exposure to the simulated oil spill, all mice in the Control Group developed alopecia which progressed during the study into severe alopecia and hyperkeratosis. By the end of the fourteenth day after exposure to the simulated oil spill, a number of the mice in the Control Group were completely hairless.

Accordingly, it may be concluded that exposure of mice to the absorbent of the invention can result in restoration of normal health very shortly after exposure to an oil spill.

EXAMPLE 6 -- CLEAN UP OF DUCKS EXPOSED TO A SIMULATED OIL SPILL

Twelve ducks (six male and six female) having a body weight in the range 1.2-1.8 kg were divided into two groups and allowed a seven day acclimatization period. One group was assigned as a Test (cleaned up) Group while the other group was assigned as a Control (noncleaned) Group. A plastic container with a surface area of 7238 cm^2 was filled with water at a temperature of 20-22°C. 1200 g of crude oil (0.17 g/cm^2 surface)

was spilled over the water surface into the container to provide a simulated oil spill. This container was emptied after each duck, and a new portion of water and oil was added for the next duck.

Each of the six ducks in the Test Group were placed into the container with the simulated oil spill. The ducks were allowed to swim in the oil spill and within a period of 2 to 4 minutes, they became disorientated and started drowning (possibly due to the anaesthetic effect of the vapours from the oil). Thus, the exposure period to the simulated oil spill was reduced to the time at which this was observed. After the exposure period, the ducks were removed from the container, the excess water/oil droplets where shaken off, and each duck was placed into a large drum with a perforated lid and 350 g of absorbent according to the invention. The container was slowly rotated for 10 minutes to allow the duck full exposure to the absorbent. Thereafter, the ducks were placed in their respective cages for further observation.

The ducks in the Control Group underwent exactly the same procedure as the ducks from the Test Group with the exception that the ducks from the Control Group were not exposed to the absorbent. Thus, after exposure to the simulated oil spill, the ducks of the Control Group were placed directly in their respective cages for further observation.

Food and water were supplied to both Groups of ducks for an observation period of 14 days after exposure to the simulated oil spill.

The ducks in both the Test and Control Groups initially lost body weight and exhibited toxic symptoms such as wings drooping, incoordination (loss of fine coordination), disorientation and lethargy. Food and water intake was diminished in both Groups.

All of the ducks in the Test Group regained body weight and appeared normal by the seventh day after exposure to the simulated oil spill.

In contrast, toxic symptoms for the ducks in the Control Group were more severe than symptoms observed for the ducks in the Test Group. The more severe toxic symptoms observed for the ducks in the Control Group resulted in the survival of three members of this Group (50%). It will be noted that the mortality rate of the Control Group was statistically significant at $p \leq 0.05$, when compared to the results obtained for the ducks in the Test Group.

On the basis of the foregoing, it may be concluded that exposure of ducks to the absorbent according to the invention marketedly increases the ducks chances for survival after exposure to a simulated oil spill.

CLAIMS:

1. A process for preparing polyethylene sorbent material useful for sorbing oil spilt on water, which comprises:

compacting small foam polyethylene pieces so as largely to destroy any closed cell foam structure thereof and to open substantially all of the foam cells thereof to interior liquid access;

and shredding the compacted foam pieces to a web-like, microcavernous network condition, effectively completely the destruction of the closed cell structure.

2. The process of claim 1 wherein the small foam polyethylene pieces are of closed cell foam structure, with a density of from 0.5-10 $lb/ft.^3$ (8.01-160.2 kg/m^3).

3. The process of claim 2 wherein the shredding of the compacted foam pieces takes place at high speed.

4. A process for selectively sorbing liquid hydrocarbons from a substrate with which the liquid hydrocarbon is incompatible, which comprises applying to the hydrocarbon and substrate mixture, a web-like, microcavernous polyethylene material prepared from foamed polyethylene pieces by a process of compacting the pieces largely to destroy the cellular structure thereof, then shredding the compacted pieces to a web-like microcavernous form.

5. The process of claim 4 wherein the hydrocarbon is crude oil.

6. The process of claim 5 wherein the hydrocarbon is heavy crude oil.

7. The process of claim 4, claim 5 or claim 6 wherein the substrate is the body surface of a mammal, a bird or a species of marine life.

8. The process of claim 5 wherein the hydrocarbon is bunker C oil.

9. The process of claim 4 wherein the substrate is water.

10. The process of claim 9 wherein the water is salt water.

11. Use of comminuted, crushed, shredded polyethylene foam as a cleanup material for oil spills at sea.

12. A sorbent material for sorbing liquid hydrocarbons, and comprising polyethylene pieces having a web-like, microcavernous network structure.

13. The sorbent material of claim 12 wherein the polyethylene pieces have an appearance under magnification substantially as shown in FIGURES 2C and 2D of the accompanying drawings.

14. The sorbent material of claim 12 wherein the polyethylene pieces comprise compacted, shredded foamed polyethylene essentially free from residual closed cells.

15. The sorbent material of claim 12, claim 13 or claim 14, essentially free from added or bound additional sorbent materials.

FIG.1.

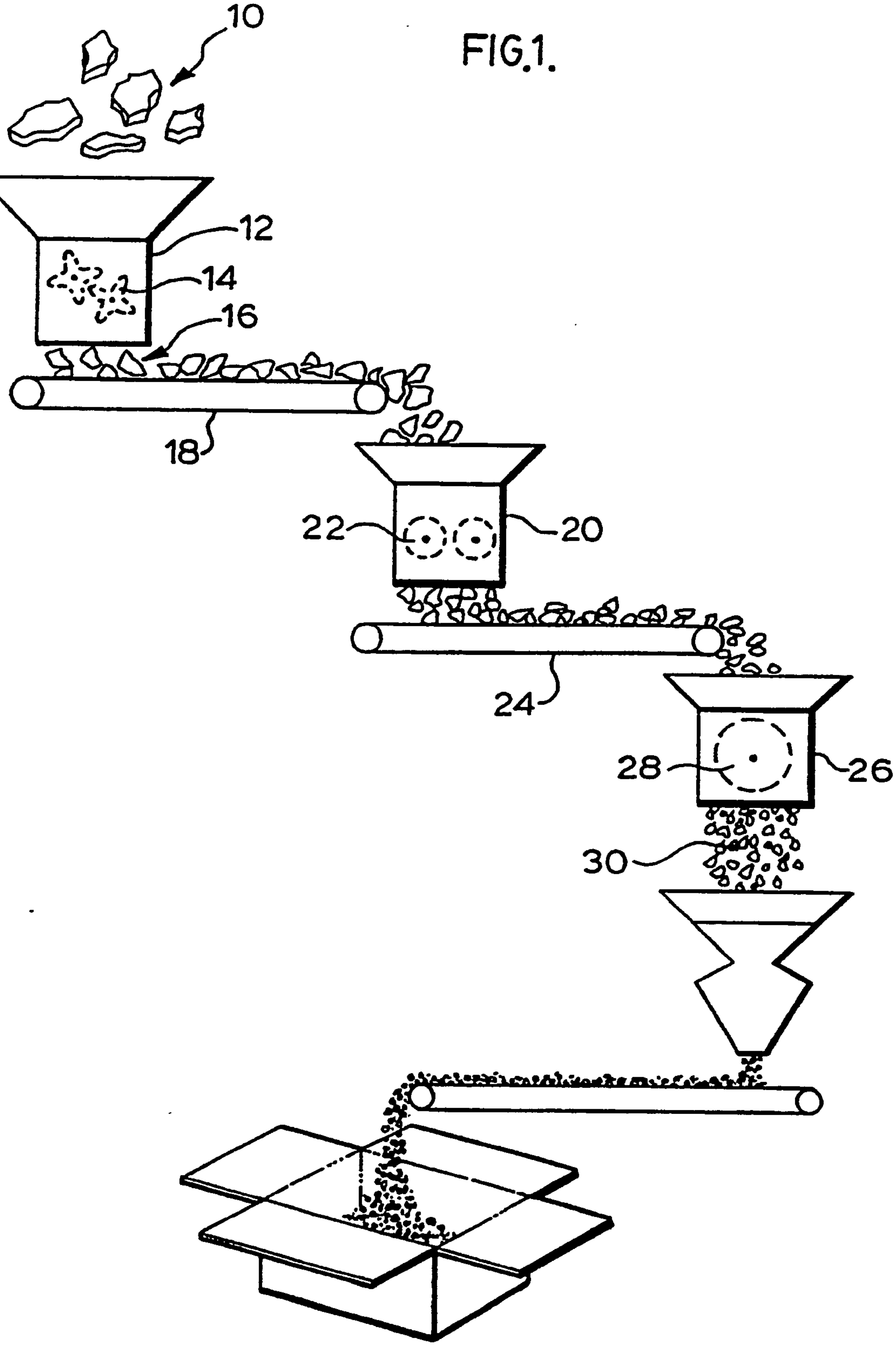


FIG. 2A


REGULAR
000002 20KV X20.0 1.50mm

FIG. 2B


CRUSHED
000003 20KV X20.0 1.50mm

FIG. 2C


SHREDDED
000004 20KV X20.0 1.50mm

FIG. 2D

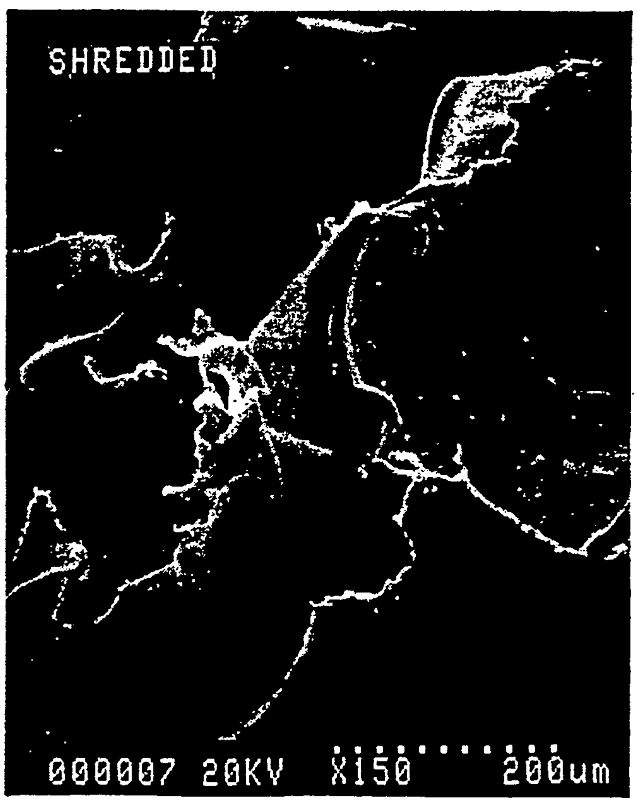

SHREDDED
000007 20KV X150 200um

FIG.3.

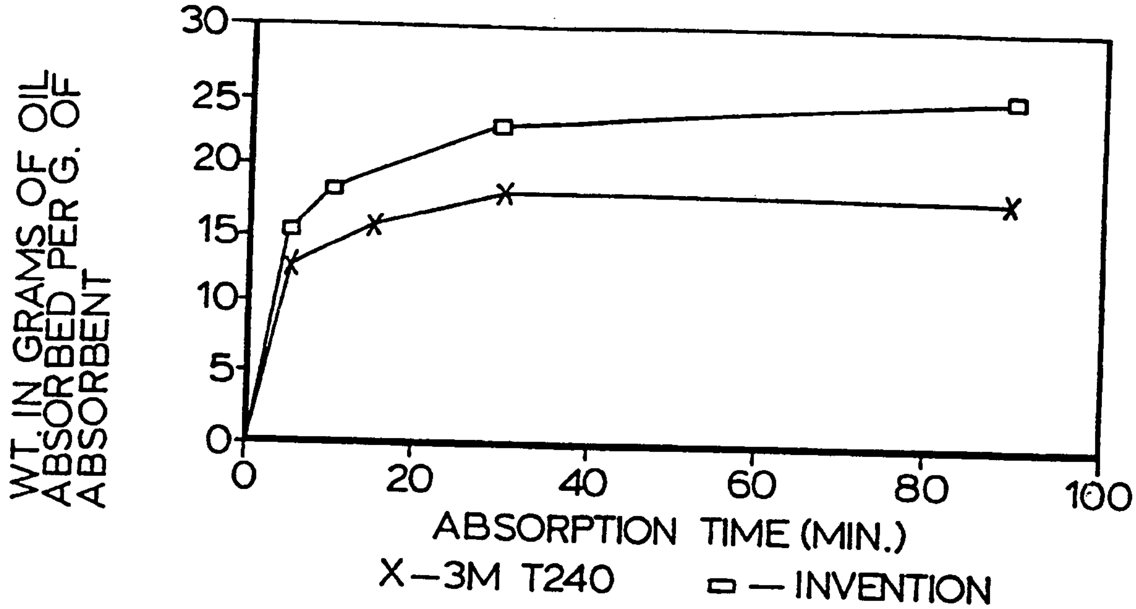
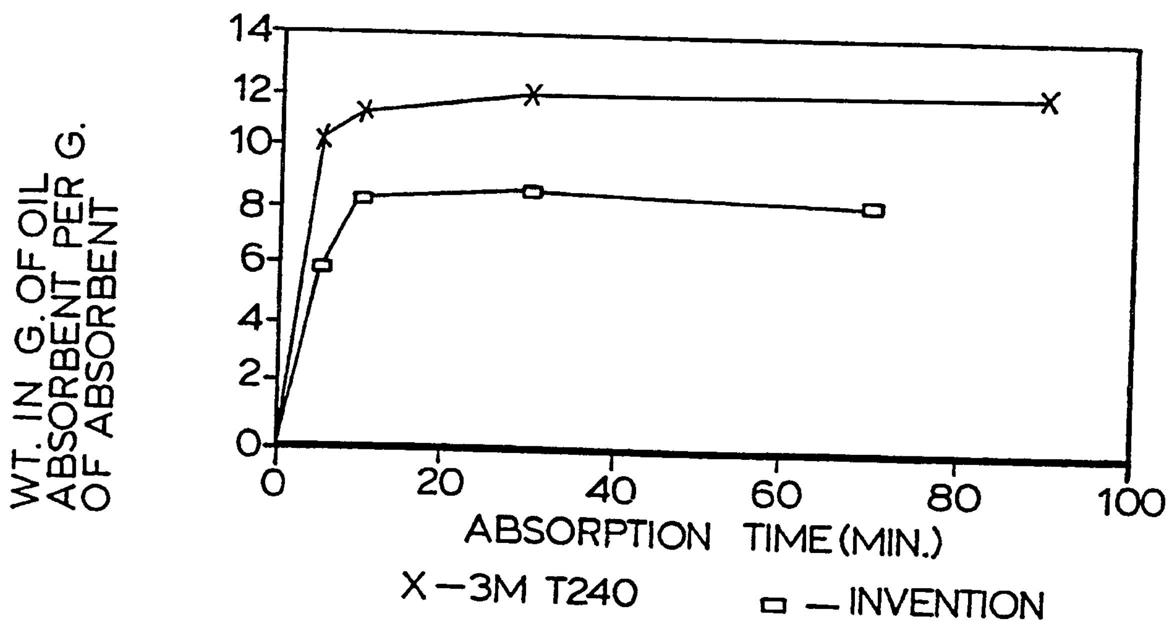

WT. IN GRAMS OF OIL ABSORBED PER G. OF ABSORBENT
30
25
20
15
10
5
0
0
20
40
60
80
100
ABSORPTION TIME (MIN.)
X—3M T240
□ — INVENTION

FIG.4.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10
8
6
4
2
0
0
20
40
60
80
100
ABSORPTION TIME (MIN.)
X—3M T240
□ — INVENTION

INTERNATIONAL SEARCH REPORT

International Application No PCT/CA 90/00166

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [6]

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC^5: B 01 J 20/26, C 02 F 1/68

II. FIELDS SEARCHED

Minimum Documentation Searched [7]

Classification System	Classification Symbols
IPC^5	C 02 F

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT [9]

Category [*]	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No. [13]
Y	US, A, 4183984 (BROWERS) 15 January 1980 see column 1, lines 5-12; column 1, line 50 - column 2, line 35; column 4, lines 31-38	1,4,9,11, 15
A		2,5,6
Y	DE, A, 1629775 (WANNER) 28 October 1971 see pages 1-2	1,4,9,11, 15
A	GB, A, 1504300 (W.E. JACOBS) 15 March 1978 see page 23, lines 6-47; page 1, line 6 - page 2, line 42	1,5-10
A	US, A, 3146192 (McCLINTOCK) 25 August 1964	

* Special categories of cited documents: [10]

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IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
31st July 1990	11. 09. 90
International Searching Authority	**Signature of Authorized Officer**
EUROPEAN PATENT OFFICE	M. Peis M. PEIS

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO. CA 9000166

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 04/09/90
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A- 4183984	15-01-80	None	
DE-A- 1629775	28-10-71	None	
GB-A- 1504300	15-03-78	AU-A- 7592374	03-06-76
US-A- 3146192		None	

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82